

AMENDMENTS TO THE SPECIFICATION

Please amend the specification as indicated hereafter. It is believed that the following amendments and additions add no new matter to the present application.

In the Specification:

Please amend the paragraph beginning on page 1, line 8:

B₁ Colloidal silica is a suspension of very small, spherical particles of amorphous (not crystalline) silica suspended in water. The material is colloidal in that the silica particles do not settle out of the solution. The silica particles generally range in size from about 8 nanometers to a maximum size of about 80 500 nanometers, beyond which the silica ceases to be colloidal and begins to settle out of solution. Colloidal silica has been used for a variety of purposes, including precision casting, as a lining for molds, as a frictionizing (non-skid) agent, and in a variety of medical and pharmaceutical uses such as toothpaste, dental castings and drug delivery systems.

Please insert on page 2, after line 19, the following paragraph taken from page 4, line 27 - page 5, line 7:

B₂ One method of producing a colloidal silica sol having a substantially uniform particle size is disclosed in US. Patent No. 3,789,009 to Irani. The sols are produced by adding simultaneously an alkali metal silicate and a cation exchange resin to a heel of water containing preformed colloidal silica particles at a pH within the range of 8-11, at a temperature of 60-150 degrees Celsius, and at a rate below that at which nucleation occurs. The cation exchange resin is added to the heel to remove the alkali metal cations from the alkali metal silicate causing the silicic acid to polymerize onto the heel nuclei to form large silica sols. Since the polymerization rate is similar for all of the uniform seed particles, the resulting larger sols also have a relatively uniform size distribution. By controlling the amount of alkali metal silicate and resin added it is possible to produce colloidal silica particles having a uniform size of between 8 nm and 200 nm. In this process, preferably the silicic acid concentration is maintained below the nucleation point to avoid the formation silica particles not sharing in the uniform size. Other processes to produce such sols are well known in the art.

Please amend the paragraph starting on p. 3, line 1 as follows:

B3 In a further preferred embodiment of the present invention, the ion exchange resin which has previously been utilized to remove potassium from the potassium silicate ~~my~~ may be utilized in the production of high purity potassium salts. To produce a very low sodium, high purity potassium salt, the ion exchange resin may be regenerated by contacting it with an acid to produce a quantity of potassium salt, which may be further purified by evaporation, crystallization and/or ultrafiltration. The low-sodium potassium salt produced is further processed by electrodialysis and/or electrolysis in order to produce a low-sodium potassium hydroxide, suitable for re-addition to the low-sodium colloidal silica to produce a very low sodium potassium hydroxide stabilized CMP silica slurry.

Please amend the paragraph starting on p. 5, line 8 as follows:

B4 The silica sols are then concentrated by either evaporation or, more preferably, ultrafiltration to recover an aqueous colloidal silica sol containing 5%-55% by weight SiO_2 SiO_2 and having a pH of between 8 and 10.5. In the preferred embodiment ultrafiltration is used to wash out contaminating sodium by replacing the aqueous solution with deionized or distilled water while simultaneously adding a small amount of ultrapure potassium hydroxide to maintain the pH. Ultrafiltration is also useful to concentrate the colloidal silica sol to a desired concentration. The resulting colloidal silica sol also optimally has a sodium concentration of less than 10 ppm sodium, but more preferably, less than 1 ppm sodium. The silica sols generally have an average particle size of 8-200 nm and preferably of from 10-80 nm and a particle size distribution with coefficient of dispersion of 0.16-0.5 and preferably 0.16 to 0.25. These sols exhibit long-term stability.

Please amend the paragraph starting on p. 6, line 20 as follows:

B5 In an alternative embodiment, the potassium sulfate may be split by subjecting the KOH solution to electrolysis in a standard two compartment cell to produce electronics grade KOH. The dialysis is achieved by circulating KOH on both sides of the cell and separated by a ~~Nafion~~ NAFION® membrane. On the anode side of the cell oxygen gas and protons are produced. On the cathode side, hydrogen gas and hydroxyl ions are the products. The hydroxyl

B6
cont
ions would combine with the potassium ions that cross the membrane to produce KOH. The membrane preferentially transports potassium over sodium because the concentration of potassium is much higher than that of sodium and also because the partition coefficient of potassium into ~~Nafion~~ NAFION® is about three to four times that of sodium. The net result is a concentrated KOH solution with very low sodium concentration.

Please amend the paragraphs starting on p. 7, lines 12, 20, and 28 as follows:

B6
A colloidal silica sol was produced generally in accordance with the methods outlined in US patent number 2,631,134 to Iler et al.- Approximately 3 liters of Purolite C-106 cation exchange resin in the hydrogen form was suspended in 10 liters of deionized water in an agitated 5 gallon vessel and heated to 95 degrees Celsius. To this was rapidly added a volume of ~~Zacsil~~ ZACSil® E200 potassium silicate sufficient to deplete the ion-exchange resin, ending up with a final pH of 8.93. In this instance, 3,950 grams of the potassium silicate solution were required. The resulting colloidal suspension had an average particle size of 10,95 nanometers in diameter and a ratio of SiO₂ to K₂O of 38.

A portion of this material was then further treated with the same type of potassium silicate and ion-exchange resin according to the method taught by US patent No. 3,789,009 to Irani. The purpose of this process is to increase the particle sizes of the silica through accretion of active silica onto a heel of smaller particles, or ~~nuclei~~ nuclei. In this fashion, the particles can be "grown" to 300 nanometers, or more, if desired. Twenty-five (25) gallons of material with an average particle size of 70 nanometers was produced. At this point, the material contained approximately 10% silica solids by weight.

The dilute sol was then concentrated, using an ultrafiltration unit, as is well known in the industry. In this fashion, the material was concentrated to approximately 35% SiO₂. When measured by atomic absorption, this suspension had a total sodium content of 83 parts per million. This included sodium in the carrier solution as well as any which may have been occluded within or adsorbed upon the silica particles themselves.

Please amend the paragraph starting on p. 8, line 22 as follows:

B7 The same process of ultrafiltration/diafiltration described in example one was used to concentrate the suspension and to dilute and remove dissolved impurities. In this case, the process started with 30 gallons of dilute sol, concentrated to about 35% solids. At this point, the sodium content was measured by AA to be 12.9 ppm. Sixty (60) gallons of diluting water was used, as described in example one, five gallons at a time. The final solution was measured at 1.2 ppm sodium content. When the reagent grade potassium hydroxide was added, the resultant 30% SiO₂ product contained 2.2 ppm sodium.

Please amend the paragraphs starting on p. 9, lines 19 and 25 as follows:

B8 Electrodialysis experiments were conducted in two separate electrodialysis cells. In the first set of experiments, a Eurodia EUR 2-C-5 stack was used. The stack had a platinized titanium anode and stainless steel cathode. The gaskets were made of an ethylene-propylene terpolymer (EPDM). The effective unit surface area in this stack is 0.2_m². Neosepta ACM anion exchange membranes, CMB cation exchange membranes and BP-1 bipolar membranes were utilized and were arranged as shown in Figure 2,

In the second set of electrodialysis experiments, an Electrosynthesis ED-I-BP stack was used. This stack also had a platinized titanium anode and stainless steel cathode. The effective unit surface area of this stack is 0.01_m². The membranes used in this stack were the same as used in the Eurodia stack and were arranged as shown in Figure 3. Most components of the stack (frames, gaskets and bipolar membranes) were soaked in a dilute KOH solution prepared from reagent grade pellets for several hours before the stack was built.

Please amend the paragraph starting on p. 10, lines 6 as follows:

B9 The electrodialysis experiments were conducted in an Electro MP cell (Electrocell AB, Sweden) equipped with nickel electrodes and a ~~Nafion~~ NAFION® 450 cation exchange membrane. This membrane is composed of a 3200 equivalent weight polymer and is typically used for KOH production. Power was supplied by a ~~Xantrex~~ XANTREX® #XFR12~100 power supply. All components of the cell (PVDF frames, EPDM gaskets, membrane) were soaked in dilute KOH solution prepared from reagent grade pellets for several hours before

B₉
cont. the cell was built. In addition, dilute KOH was circulated through both sides of the cell for several hours to remove any traces of contamination. Both anolyte and catholyte reservoirs were HDPE and were connected to the cell via ~~teflon~~ TEFLON® tubing. The starting anolyte solution was prepared from reagent grade KOH pellets.

Please amend the paragraph starting on p. 10, line 22 as follows:

B₁₀ A typical ED experiment was conducted as follows; 3.5 liters of a saturated solution was first prepared by dissolving K₂SO₄ crystals into deionized water. The resultant solution was then filtered through a 5 micron ~~millipore~~ MILLIPORE® filter before being introduced into the feed reservoir. When the high sodium saturated solution was used, it, too, was filtered through a 5 micron filter prior to use. The initial base consisted of 500 ml deionized water. The initial acid solution and electrode rinse both consisted of one liter of nominally 0.5M sulfuric acid. The electrode rinse was reused for subsequent experiments.

Please amend the paragraphs starting on p. 11, lines 18 and 23 as follows:

The current efficiency for KOH production for all of the ED experiments (see FIG. 4) varied between 82% and 87%, with the main inefficiency being the back migration of hydroxyl ions across the cation exchange membrane. It is also possible that proton transport from the acidified feed compartment contributed to the inefficiency. The strength of KOH produced varied between 2.33M (11.6% w/w) ~~an~~ and 3.55M (17.2% w/w).

B₁₁ The current efficiency for acid production averaged 78% for the two experiments with the Eurodia stack. The current efficiencies obtained with the ED-1BP stack started at 76% for the first experiment then dropped down to 50% for the third and final experiment. In all cases loss in current efficiency is due primarily to the back migration of proton from the acid compartment across the anion exchange membrane (the pH of the feed dropped in all experiments). It is also possible that part of the inefficiency is due to the diffusion of sulfuric acid molecules out of the acid compartment into the feed compartment. The initial efficiencies observed are consistent with what can be expected for this type of anion exchange membrane. The deterioration of the acid current efficiency should be evaluated

B11
cont
over a longer time period before drawing any final conclusions; however, it is possible that the lower current efficiency (CE) in the last run is a result of using the mother liquor which may contain a higher level of impurities.

Please amend the paragraph starting on p. 12, line 3 as follows:

B12
In four of the five runs conducted, sulfate concentrations detected in the KOH product varied between 1.1 mM (100 ppm) and 2.1 mM (200 ppm) corresponding to diffusion coefficients of $1.9 - 2.8 \times 10^{-12} \text{ m}^2/\text{sec}$ and an average of $2.4 \times 10^{-12} \text{ m}^2/\text{sec}$. One of the five runs had a relatively higher rate of diffusion (360 ppm and $6.0 \times 10^{-12} \text{ m}^2/\text{sec}$). This result may have been due to experimental error. Note that the level of transport is consistent with that normally seen for bipolar membranes.

Please amend the paragraphs starting on p. 12, lines 14, 23, 26, and 31 as follows:

B13
The ratio of partition coefficients for K vs. Na is a measure of the membrane's preference for transporting one species over the other. Measurements of the average $K^+ : Na^+$ ratios in the feed and the final ratios in the KOH product indicated that they were very consistent for all three runs using the ED-1-BP stack and resulted in an average partition coefficient ratio of 0.93 for K vs. Na (~~FIGs~~ FIG. 5 and FIG. 6), indicating that the membrane shows very little preference for either species. This is lower than found for ~~Nafion~~ NAFION® membranes but is very consistent with previous experience gained at Electrosynthesis Company (ESC). The results ~~obtains~~ obtained when the a K_2SO_4 solution with a high Na K_2SO_4 solution content was used (Run 484-31) are similar ~~and indicate~~. This indicates that the ratio was independent of the relative concentrations of the two species.

Two electrolysis runs were performed, one at $200 \text{ mA}/\text{cm}^2$ and the other at $350 \text{ mA}/\text{cm}^2$. The results (see ~~FIGs~~ FIG. 7 and FIG. 8) showed that cathodic current efficiencies were consistently high at 98.6% and 98.7%, producing KOH at 4.37M (20.4%) and 7.45M (32%). In ~~Nafion~~ NAFION® membranes of 1200 equivalent weight polymer the K:Na coefficient ratio is 3.97; : 1.22 or 3.25 times that of sodium. Analysis of the anolyte and catholyte

solutions before and after electrolysis showed that the ratio of K vs. Na increased significantly, indicating that a much purer product was formed, and resulting in an average partition coefficient ratio of 3.75 for K vs. Na into the ~~Nafion~~ NAFION® membrane.

B3
cont
Based on the partition coefficient ratios obtained for the electro dialysis and electrolysis experiments, it is possible to calculate the maximum concentration of Na impurity that must be present in the starting saturated K_2SO_4 solution. A calculated starting maximum Na concentration of 6.3 ppm Na in 0.7M (13.4%) K_2SO_4 must be achieved in order to produce a 9.27M (40% w/w) solution of KOH with a maximum of 10 ppm Na impurity. This level was achieved in saturated solutions prepared from crystals supplied by Chemical Products Corporation (CPC).

Please amend the paragraphs starting on p. 13, lines 4, 12, 17, and 22 as follows:

B14
It should also be noted that in the final electro dialysis experiment conducted with 5 solution made from crystal, we were able to produce a KOH solution of sufficient purity required for further electrolysis to produce 9.27M (40% w/w) KOH as specified above. These experiments demonstrate that potassium sulfate solutions can be split into sulfuric acid and potassium hydroxide by electro dialysis using a three compartment cell incorporating bipolar membrane technology. KOH solutions of up to 3.55M (17.2% w/w) can be produced with very good current efficiency (86%) and low sulfate contamination of 1.6M (133 ppm) at peak operating current densities of up to 200 mA/cm².

There was virtually no preference for transport of potassium vs sodium across the ~~Neosepta~~ NEOSEPTA® CMB cation exchange membrane. The ratio of K vs. Na in the feed was very similar to that found in the KOH product. The average partition coefficient ratio for K vs. Na was 0.93:1. This is consistent with previous experience gained at ESC. Sulfuric acid at concentrations of up to 1.3 5M can be produced at current efficient of 76%. With regard to the electrolysis of reagent grade dilute KOH solutions to produce high purity KOH, ~~Nafion~~ NAFION® membranes demonstrate a definite preference for the transport of potassium vs. sodium. In the experiments performed, up to 7.5M (32% w/w) KOH was made

at very high current efficiency of 98.7% and significantly increased purity. The average partition coefficient ratio of K vs. Na was 3.75:1.

B14
cont.

Using the ratios of partition coefficients obtained during our electrodialysis and electrolysis experiments, we calculate that in order to achieve a final 9.27M (40% w/w) product with 10 ppm Na contamination, a starting saturated K_2SO_4 solution of 0.7M (13.4%) must contain 6.3 ppm or less of Na.

In the Drawings:

Please replace drawing sheets 1-7 (showing Figs. 1-7) with the newly-submitted figures attached herewith on separate sheets.